Determination of Oven Moisture in Tobacco

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A comparison of mechanical convection and gravity convection oven-drying of tobacco was made on 12 tobacco samples, six with high and six with low moisture content. Data show that better interlaboratory precision was obtained by drying in a mechanical convection oven. The term "apparent moisture" is used because oven drying also causes loss of volatile organic material. The magnitude of this loss is shown to be from 0.25 to 1.5%, depending on the type and treatment of the tobacco. The forced draft oven method is recommended for adoption as official, first action.

When moisture is determined in tobacco by oven methods, volatiles other than water are lost, and this loss causes the "moisture" figures to be higher than the true values. This fact is recognized by the tobacco industry, and although the data obtained are not accurate moisture values they can be used to correct other analytical data to a reasonably uniform moisture-free basis. In this report "moisture" refers to the loss in weight of tobacco when dried in an oven or over desiccant.

The methods used in this study, conducted several years ago by the Analytical Methods Committee of the Tobacco Chemists' Research Conference, were designed for ground analytical samples and not for bulk leaf, strip, or scrap material.

Twelve samples of tobacco were prepared by the American Tobacco Company for this study. Six different tobaccos were ground to pass a 1 mm screen, and each was divided into two lots; then one lot was conditioned to produce a high moisture sample and the other a low. The tobacco samples and designations were as follows:

	Moisture Level	
	Low	High
Lug, flue-cured	1A	1B
Leaf, flue-cured	2A	2B
Lug, burley	3A	3B
Leaf, burley	4A	4B
Cased, blended, cigarette	5A	5B
Cigar, filler	6A	6B

Three methods were tested. In addition each collaborator was asked to determine the moisture by his own procedure if it differed from those under test.

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Method I

Apparatus

(a) *Drying oven.*—Forced-draft, regulated to 99.5 ± 0.5°. Suggested dimensions: 19 × 19 × 19″. Approx. oven settings: fresh air intake vent ⅛ open; air control damper ¼ open; air exhaust vent ⅓ open.

(b) *Moisture dish.*—Al, diam. 45–65 mm, depth 20–45 mm, with tight fitting cover.

Determination

Weigh accurately ca 5 g sample (ground to pass 1 mm or finer screen) into weighed moisture dish and place uncovered dish in oven.

Do not exceed 1 sample/10 sq. in. shelf space, and use only 1 shelf. Dry 3 hr at 99.5 ± 0.5°; remove from oven, cover, and cool in desiccator to room temp. (ca 30 min.). Reweigh to nearest 1 mg and calc. % moisture = (wt before − wt after drying) × 100/wt sample.

Method II

Proceed as in method I, except use convection oven. Limit number of dishes to keep 50% shelf space free.

Method III

Apparatus

(a) *Desiccator.*—Standard 10–12″ laboratory desiccator containing not < 300 ml fresh 95% H_2SO_4.

(b) *Moisture dish.*—*See* method I.

Determination

Weigh 2 g sample to nearest mg in moisture dish, place in desiccator with cover removed, and let stand 9 days at 30°C. Do not exceed 8 samples/desiccator and do not stack dishes. After 9 days remove from desiccator, cover, and reweigh immediately to nearest mg. Calculate % moisture.

Method IV

If collaborator's usual procedure differed from methods I, II, or III, he was asked to determine moisture by his method and report the results.

Results and Recommendation

The results obtained by the various collaborators and methods are shown in Table 1. Since the method is empirical and because we recognize that the results are higher than the true value, the main consideration is the between-laboratory precision. This precision is shown by the variance figure in the last two columns. The next to last column shows the variance for all data; the last column shows the variance for method I (B series), with the data from Collaborator 102 excluded. The data from this collaborator for the first 6 samples were randomly distributed about the average, the value for sample 1B was slightly higher than the average for this sample, and all values were low by 0.5–1% for the last 5 samples, indicating a probable malfunction of apparatus. Since such a distribution of values would occur normally less than 10% of the time, the second set of variances was calculated and is believed to be a truer representation of the precision of method I. Comparison of variances for methods I and II showed that in all but one case the variance for method I was lower than that for method II. For 5 of the 12 samples the difference was critical (F test) at the 10% level.

Based on interlaboratory precision, method I is the method of choice. This is in general agreement with laboratory practice since most tobacco research laboratories now use a mechanical convection oven method with only slight, if any, differences from method I.

Method III, the desiccator procedure, was included in the study to provide some indication as to the magnitude of the difference between the oven "moisture" figure and the true value. The between-laboratory agreement for this method was very poor. However, three laboratories (103, 104, and 108) were in reasonably close agreement and obtained higher values than the other collaborators. The differences between the median value for each sample in these three sets of data and the median value obtained by method I for the same sample give an indication of the amount of volatiles other than water lost on oven drying (Table 2). Table 2 also shows that the difference for the A and B samples of each tobacco was

The recommendation of the Associate Referee was approved by the General Referee and by Subcommittee A, and was adopted by the Association. See *This Journal*, **49**, 172–175 (1966).

Table 1. Collaborative results for moisture in tobacco

Sample	Legend	Code Numbers of Collaborators 101	102	103	104	105	106	107	108	Av. % Moisture	Variance	
						Method I						
1A	Lug, flue	5.57	5.30[b]		5.60			5.67	5.67	5.56	0.0234[c]	
2A	Leaf, flue	5.31	5.45		5.45			5.21	5.44	5.37	0.0117	
3A	Lug, burley	5.49	5.29		5.51			5.29	5.59	5.43	0.0187	
4A	Leaf, burley	4.69	4.64		4.64			4.66	4.72	4.67	0.0073	
5A	Cased, blended	4.26	4.36		4.36			4.38	4.50	4.37	0.0012[d]	
6A	Cigar, tobacco	5.24	5.41		5.30			5.34	5.35	5.33	0.0143[d]	
1B	Lug, flue	11.55	11.75		11.79			11.51	11.73	11.67	0.0159	0.0151
2B	Leaf, flue	11.00	10.50		11.06			11.01	11.17	10.95	0.0673	0.0061[d]
3B	Lug, burley	9.97	9.44		10.39			9.95	10.21	9.99	0.1215	0.0452
4B	Leaf, burley	10.33	9.67		10.67			10.53	10.63	10.37	0.1687	0.0231
5B	Cased, blended[a]	12.60	12.16		12.77			12.75	12.99	12.65	0.0956	0.0258[d]
6B	Cigar, tobacco	10.56	9.91		10.82			10.55	10.77	10.52	0.1318	0.0191[d]
						Method II						
1A	Lug, flue	5.20	5.50	5.47		5.3[b]	5.47	5.66	5.75	5.48	0.0361	
2A	Leaf, flue	5.09	5.26	5.21		5.2	5.56	5.36	5.07	5.25	0.0284	
3A	Lug, burley	5.26	5.32	5.39		5.4	5.57	5.15	5.59	5.38	0.0253	
4A	Leaf, burley	4.52	4.56	4.45		4.5	4.74	4.73	4.26	4.54	0.0277	
5A	Cased, blended	3.80	4.20	3.92		4.1	4.10	4.33	5.11	4.22	0.0275	
6A	Cigar, tobacco	5.04	5.30	5.17		5.2	5.27	5.57	5.89	5.35	0.0687	
1B	Lug, flue	11.09	11.41	11.34		11.2	11.68	11.59	11.76	11.43	0.0623	
2B	Leaf, flue	10.80	10.79	10.83		10.8	11.27	11.11	10.81	10.92	0.0374	
3B	Lug, burley	9.98	9.76	9.79		9.8	10.08	10.19	10.19	9.97	0.0375	
4B	Leaf, burley	9.84	10.11	10.19		10.1	10.18	10.59	10.14	10.16	0.0490	
5B	Cased, blended[a]	12.47	12.21	11.65		12.6	12.42	12.61	13.62	12.51	0.3483	
6B	Cigar, tobacco	10.25	10.29	10.42		10.6	10.75	11.02	11.27	10.66	0.1460	
						Method III						
1A	Lug, flue	3.47	3.15	4.78	4.66	4.2		4.10	4.49	4.12		
2A	Leaf, flue	3.33	3.18	4.84	4.51	4.2		3.70	4.42	4.03		
3A	Lug, burley	3.74	3.22	5.43	4.94	4.3		3.88	5.01	4.36		
4A	Leaf, burley	3.19	2.34	4.46	3.92	3.6		3.12	3.82	3.49		
5A	Cased, blended	2.13	1.87	3.08	2.90	2.6		2.25	2.79	2.52		
6A	Cigar, tobacco	3.98	2.61	5.41	4.02	4.2		3.70	4.88	4.11		
1B	Lug, flue	9.39	8.66	10.76	10.32	10.2		9.82	10.66	9.97		
2B	Leaf, flue	9.03	8.37	10.24	10.03	9.6		9.48	10.18	9.63		
3B	Lug, burley	8.70	7.22	9.50	9.16	9.0		8.63	9.59	8.83		
4B	Leaf, burley	8.67	7.41	9.68	9.38	9.3		8.80	9.74	9.00		
5B	Cased, blended[a]	10.54	9.74	11.27	10.93	10.8		10.65	11.19	10.73		
6B	Cigar, tobacco	9.40	7.16	10.30	9.34	9.4		8.75	10.31	9.24		
						Method IV						
										RANGE		
1A	Lug, flue	6.45		4.26	5.58		5.89	6.73	5.64	2.47		
2A	Leaf, flue	6.22		4.02	5.42		5.69	5.89	5.25	2.20		
3A	Lug, burley	6.06		4.37	5.47		5.83	6.41	5.34	2.04		
4A	Leaf, burley	5.26		3.36	4.59		4.93	5.78	4.65	2.42		
5A	Cased, blended	5.47		2.57	4.36		4.98	5.49	4.39	2.92		
6A	Cigar, tobacco	5.84		4.51	5.29		5.56	6.58	5.25	2.07		
1B	Lug, flue	12.46		10.38	11.64		11.79	12.53	11.60	2.15		
2B	Leaf, flue	11.88		9.97	11.13		11.36	11.62	10.95	1.91		
3B	Lug, burley	10.57		9.19	10.13		10.38	10.97	9.82	1.78		
4B	Leaf, burley	11.02		9.25	10.49		10.62	11.43	10.33	2.18		
5B	Cased, blended[a]	13.81		10.84	12.76		13.39	14.14	12.66	3.30		
6B	Cigar, tobacco	11.15		9.79	10.70		10.79	11.66	10.44	1.87		

[a] Cigarette tobacco.
[b] Regular method for this collaborator.
[c] Data from collaborator 102 omitted.
[d] Variances for Method I significantly less than those for Method II (10% level).

reasonably consistent. Comparison of the values by method I with those obtained by GLC (gas-liquid chromatography) would probably give a truer measure of volatile organic material lost during oven drying. However, GLC methods for moisture in tobacco were not developed when this study was made.

Table 2. Comparison of median moisture values for methods I and III

Sample	Medians I[a]		III		Differences	Δ_B
1A	5.60	–	4.66	=	0.94	1.07
2A	5.44	–	4.51	=	0.93	0.83
3A	5.49	–	5.01	=	0.48	0.45
4A	4.66	–	3.92	=	0.74	0.95
5A	4.36	–	2.90	=	1.46	1.56
6A	5.34	–	4.88	=	0.46	0.26
1B	11.73	–	10.66	=	1.07	
2B	11.01	–	10.18	=	0.83	
3B	9.95	–	9.50	=	0.45	
4B	10.63	–	9.68	=	0.95	
5B	12.75	–	11.19	=	1.56	
6B	10.56	–	10.30	=	0.26	

[a] Median of 3 highest sets of values.

Because method I gave good precision on all types of tobacco tested at both high and low levels of moisture and was more precise than method II, and because it is the method most used in the industry today, it is recommended that method I, the forced draft oven method, be adopted as official, first action.

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